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FORM PTO-1 (REV-11-98)	190 U.S. DEPA	RTMENT OF COMMERCE PATENT AND TRADEMARK OFFICE	ATTORNEY'S DOCKET NUMBER
T	RANSMITTAL LETTEI	R TO THE UNITED STATES	Mo-5884/LeA 32,873
1	DESIGNATED/ELECT	TED OFFICE (DO/EO/US)	US APPL 019 / 646450
	CONCERNING A FILI	NG UNDER 35 U.S.C. 371	To be Assigned
1	ATIONAL APPLICATION NO.	INTERNATIONAL FILING DATE	PRIORITY DATE CLAIMED
	P99/01537	03 March 1999 (3.03.99)	18 March 1998 (18.03.98)
	OF INVENTION SOOT GR	ANULES	
APPLIC Gunter L	ANT(S) FOR DO/EO/US inde; Uwe Hempelmann, Manfred	Eitel	
		tes Designated/Elected Office (DO/EO/US) the foll-	owing items and other information:
		ms concerning a filing under 35 U.S.C. 371.	
2. 📙		ENT submission of items concerning a filing under	
3. X 4. X	examination until the expiration of	onal examination procedures (35 U.S.C. 371(f)) at a f the applicable time limit set in 35 U.S.C. 371(b) at I Preliminary Examination was made by the 19th m	and PCT Articles 22 and 39(1).
5. X	A copy of the International App	plication as filed (35 U.S.C. 371(c)(2))	
1		h (required only if not transmitted by the Inter	national Bureau).
		by the International Bureau.  application was filed in the United States Reco	-init Office (DOLLE)
6. X	is not required, as the	application was filed in the United States Reco	
7.		he International Application under PCT Articl	
	F-7	ith (required only if not transmitted by the Inte	
1	b. have been transmitted	by the International Bureau.	,
1	c have not been made;	however, the time limit for making such amen	dments has NOT expired.
1 _	d. have not been made a	nd will not be made.	
8	A translation of the amendmer	ats to the claims under PCT Article 19 (35 U.S	.C. 371(c)(3)).
9. X	An oath or declaration of the i	nventor(s) (35 U.S.C. 371(c)(4)).	
10.	A translation of the annexes to (35 U.S.C. 371(c)(5)).	the International Preliminary Examination Re	eport under PCT Article 36
Items	11. to 16. below concern docum	nent(s) or information included:	
II.	An Information Disclosure Sta	tement under 37 CFR 1.97 and 1.98.	
12. X	An assignment document for t	recording. A separate cover sheet in compliance	ee with 37 CFR 3.28 and 3.31 is included.
13. X	A FIRST preliminary amendm	ent.	OLPE
	A SECOND or SUBSEQUEN	T preliminary amendment.	
14	A substitute specification.		SEP 1 4 2000 1
15.	A change of power of attorney	and/or address letter.	CHI & TRADENSEN OF
16 X	Other items or information:	Preliminary Amendment w/Abstract; PTO Form 1449 and references listed	therein
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		PTO USE ONLY
BASIC NATIONAL FEE (37 CFR 1.492 (a) (1) - (5))		
Neither international preliminary examination fee (37 CFR 1.482) nor international search fee (37 CFR 1.445(a)(2)) paid to USPTO		
and International Search Report not prepared by the EPO or JPO	101	- E
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International preliminary examination fee paid to USPTO (37 CFR 1.482)		
and all claims satisfied provisions of PCT Article 33(1)-(4)		
ENTER APPROPRIATE BASIC FEE AMOUNT = \$	840.00	
Surcharge of \$130.00 for furnishing the oath or declaration later than 20 30 smonths from the earliest claimed priority date (37 CFR 1.492(c)).		
CLAIMS NUMBER FILED NUMBER EXTRA RATE		
Total claims 15 -20 = 0 X \$18.00 \$	0.00	
Independent claims 2 -3 = 0 X \$78.00 S	0.00	
MULTIPLE DEPENDENT CLAIM(S) (if applicable) +\$260.00 \$  TOTAL OF ABOVE CALCULATIONS = \$		
	840.00	
Reduction of 1/2 for filing by small entity, if applicable. A Small Entity Statement must also by filed (Note 37 CFR 1.9, 1.27, 1.28).	0.00	
SUBTOTAL = \$	840.00	
Processing fee of \$130.00 for furnishing the English translation later than 20 30 5 months from the earliest claimed priority date (37 CFR 1.492(f)).		
TOTAL NATIONAL FEE = \$	840.00	
Fee for recording the enclosed assignment (37 CFR 1.21(h)). The assignment must be accompanied by an appropriate cover sheet (37 CFR 3.28, 3.31). \$40.00 per property	40.00	
TOTAL FEES ENCLOSED = \$	880.00	
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a. A check in the amount of \$ to cover the above fees is enclosed.		
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PATENT APPLICATION Mo5884 LeA 32,873

## IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

APPLICATION OF

GÜNTER LINDE ET AL

SERIAL NUMBER: TO BE ASSIGNED

FILED: HEREWITH

TITLE: SOOT GRANULES

PCT/EP 99/01537

SEP 1 4 2000

SEP 1 4 2000

### PRELIMINARY AMENDMENT

Assistant Commissioner for Patents

Washington, D.C. 20231

Sir:

Prior to the examination of the subject application, kindly amend the application as follows:

### IN THE SPECIFICATION:

At page 1, line 1, and the Abstract page, change the title to read --SOOT GRANULES--.

At page 1, line 2 insert the heading -- BACKGROUND OF THE INVENTION --.

At page 3, line 1 insert the heading --SUMMARY OF THE INVENTION--.

At page 3, line 8 insert the heading --DETAILED DESCRIPTION OF THE INVENTION--.

### IN THE CLAIMS:

Cancel Claims 1- 19.

"Express Mail" mailing label number EL598329242US

Date of Deposit September 14, 2000

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PATENT TRADEMARK OFFICE



Kindly add the following Claims 20-34:

- -20. Compacted carbon black pellets having a relative color intensity, based on the uncompacted powder, of greater than 100%.
- 21. The pellets of Claim 20 wherein the pellets have an average particle size of 0.3 to 2 mm and a quotient of density and bulk density between 3.0 and 10.
- The pellets of Claim 20 wherein the pellets comprise one or more binders and/or dispersants in a total quantity of 0.1 to 25 wt.%, based on the weight of the pellets.
- The pellets of Claim 22 wherein the binder comprises a liquid polyol, polyether, polyester, oil, water or an aqueous solution of a polymeric salt or molasses.
- 24. The pellets of Claim 22 wherein the dispersant comprises a lignin sulfonate or a naphthalene/formaldehyde condensation product.
- 25. A process for the production of carbon black pellets which comprises compacting carbon black in the presence of an auxiliary substance and comminuting it to pellets having an average particle size of 0.3 to 2 mm and a quotient of density and bulk density between 3.0 and 10.
- 26. The process of Claim 25 comprising performing the compaction two or more times
- 27. The process of Claim 25 comprising performing the compaction two or more times at different compressive forces in each compaction stage.
- 28. The process of Claim 25 comprising using compression forces of between 1 and 100 kN/cm
- The process of Claim 25 comprising performing the compactation by screws, rollers, die presses or extruders.
- 30. The process of Claim 25 wherein the auxiliary substance is one or more binders and/or dispersants used in total quantity of 0.1 to 25 wt.%, based on the weight of the pellet.
- The process of Claim 30, wherein the binder comprises a liquid polyol, polyether, polyester, oil, water or an aqueous solution of a polymeric salt or molasses.

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- 32. The process of Claim 30 wherein the dispersant comprises a lignin sulfonate and a naphthalene/formaldehyde condensation product.
- 33. The process of Claim 25 comprising coating the resulting pellets with a thin layer of wax, polyether, polyolefin or polyvinyl alcohol.
- The process of Claim 33 comprising adding a preservative and/or fragrance to the wax, polyether, polyolefin or polyvinyl alcohol.--

### REMARKS

Claims 1-19 have been cancelled and replaced by Claims 20-34. These claims are supported by the original claims.

An early action of the merits is respectfully requested.

Respectfully submitted,

GÜNTER LINDE UWE HEMPELMANN MANFRED EITEL

By Momas W. Kry Thomas W. Roy

Attorney for Applicants Reg. No. 28,582



Bayer Corporation 100 Bayer Road Pittsburgh, Pennsylvania 15205-9741 (412) 777-2345 FACSIMILE PHONE NUMBER: (412) 777-5449 /vit/DP0095 U 9 / 6 4 6 4 5 0 Le A 32 873 /PB/NT/V23.02.1999-Foreight 30 Rec'd PCT/PTO 1 4 SEP 2000

Carbon black pellets

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This invention relates to a process for the production of compacted carbon black pellets for various applications.

- 1 -

By virtue of their small particle size of  $0.01\,\mu m$  to  $20\,\mu m$ , colouring materials are known to have a strong tendency to dust and to be very difficult to dispense due to the strong adhesive forces between the particles. This is overcome by pelletising these powders before use. However, pelletisation often also results in a reduction in dispersibility as the stability of the pellets is usually increased by binders in order to improve transport characteristics. As a result, pigment pellets frequently exhibit lower initial colour intensity, such that, for a given dispersing time, the pellets develop a less intense colour than when colouring with powders. The desired advantages of absence of dust and good dispensability have thus resulted in considerable efforts to obtain readily dispersible pellets for pigments too.

This applies in particular to carbon blacks which, due to the small particle size and low bulk density thereof, have always previously undergone compaction as dry powders by "degassing" between vacuum rollers or by "beading" in rotating drums. However, compaction between rollers does not yield pellets and dry pulverisation results in agglomeration of the flocculent carbon black into large spherules of up to a few millimetres in diameter. In wet beading, the carbon blacks are pelletised in beading machines using water and possibly binders and then dried. The production of carbon black pellets is described in *Ullmann's Encyclopedia of Industrial Chemistry*, fifth edition, volume A 5, page 148.

US-A 4 946 505 describes the production of carbon black and pigment pellets for colouring concrete which are obtained by spray pelletisation. The disadvantage of spray pelletisation is that it is necessary to prepare an aqueous slurry of carbon black

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and vaporisation of the water entails considerable drying costs. US-A 4 946 505 explicitly excludes compaction and briquetting processes.

DE-A 43 36 548 and DE-A 43 45 168 describe carbon black pellets which are produced with the addition of considerable quantities of water by means of an annular die press, subsequent rounding and drying. The resultant pellets contain less than 1% hinder

EP-A 0 370 490 describes carbon black flakes for printing inks which are produced by compacting carbon black under a low pressure. The material still contains dusting fractions. It is explained that dispersibility suffers if elevated pressure is used.

EP-A-0 802 241 discloses carbon black pellets which have a relative colour intensity relative to the powder on which they are based of at most 100%.

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The processes hitherto available either do not yield a satisfactory material or are unfavourable in energy terms due to post-drying.

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The object thus arose of providing carbon black pellets which combine mechanical stability with elevated colour intensity as well as a technically simple, low cost process which yields readily dispersible, low dusting earbon black pellets which are stable in transport.

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This object is achieved by the carbon black pellets according to the invention: the carbon black powder is compacted to such an extent with the addition of auxiliary substances and subsequently pelletised that the quotient of pycnometric density and bulk density is between 3.0 and 10, preferably between 3.5 and 8. Despite the compaction, the resultant pellets surprisingly exhibit greater colour intensity than the powders on which they are based.

The present invention accordingly also provides a process for the production of carbon black pellets, in which carbon blacks are compacted with auxiliary substances, in a preferred embodiment twice or more, and comminuted to yield pellets, wherein the pellets generally have an average particle size of 0.3 to 2 mm, preferably of 0.5 to 1.0 mm. The quotient of pycnometric density and bulk density of the pellets obtained according to the invention is between 3.0 and 10, preferably between 3.5 and 8.

Compressive forces preferably of between 1 and 100 kN/cm, particularly preferably of between 10 and 30 kN/cm, are used in the compacting stages. The compressive forces may be identical in all the compacting stages, but preferably differ. The product thickness achievable with these compaction units is preferably 1 to 5 mm, in particular 1 to 3 mm. Suitable compaction units are, for example, screws, rollers, die presses, extruders with or without evacuation apparatus.

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The pelletisation proceeding after the compaction stages is preferably performed by screen pelletisation. In this case, the fines fraction may be directly separated by screening and recirculated.

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It may also be advantageous to increase the size of the fines fraction by pelletisation in a subsequent stage, preferably, for example, by post-rolling on a rotary table or in a rotating drum.

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Adding liquid auxiliary substances increases throughput and pressure under given compaction conditions, such that the resultant pellets become stronger.

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Particle size distribution measurements show that the use of higher compressive forces clearly results in a breakdown of the carbon black structures. It has accordingly been observed that the pellets produced according to the invention exhibit a greater colour intensity than the corresponding powder mixtures. The

application of relatively high pressures and/or repeated compaction also results in greater pellet stability and surprisingly according to the invention simultaneously greater colour intensity.

5 The particle sizes of the pellets are not essential to the invention; they are generally from 0.3 to 2 mm, preferably from 0.5 to 1.0 mm. The quotient of pycnometric density and bulk density is between 3.0 and 10, preferably between 3.5 and 8.

One or more binders and/or dispersants are used as auxiliary substances. The auxiliary substances may be solid or liquid. It is preferred to use liquid binders from the group comprising polyols, polyethers, polyesters, oils, water and aqueous solutions of polymeric salts or molasses. A dispersant from the group comprising lignin sulfonates and naphthalene/formaldehyde condensation products is also preferred

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The total quantity of auxiliary substances is usually 0.1 wt.% to 25 wt.%, preferably 1 to 20 wt.% and particularly preferably 3 to 15 wt.%.

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The stability of the resultant pellets may subsequently be increased in a further stage by coating them with a thin layer of a material from the group comprising waxes, polyethers, polyesters, polyelefins and polyvinyl alcohols. Preservatives and/or fragrances may additionally be added.

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The process according to the invention not only yields free-flowing pellets, but the relative colour intensity in concrete is higher than would be expected on the basis of the quantity of starting material used in powder form.

The carbon black pellets according to the invention are thus particularly suitable for colouring substrates, in particular concrete, asphalt, plastics, as well as paints and lacquers.

### Method for assessing the dispersibility of pellets in building materials

Dispersibility in cement mortar (colour intensity in mortar prisms) of the pellets is determined relative to the powder standard.

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### Equipment:

Toni mixer with electronic control

Mixing container (from Toni-Technik)

Colorimeter (Minolta CR 310); illuminant C;  $0^{\circ}$  diffuse with gloss, 8 cm illumination aperture

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### Mortar formulation:

1200 g 0.2-1 mm quartz sand

600 g 1-2 mm quartz sand

200 g limestone flour (5 wt.% screening residue on 90  $\mu m$  screen)

500 g white cement (Dyckerhoff)

175 g water (water/cement ratio = 0.35)

6 g pigment pellets

### 20 Method:

All the mix components are introduced into the mixing container initially without water and premixed while dry. Water and then cement are subsequently added and the mix stirred for 100 seconds.

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This mix is placed in a steel die and compression moulded at approx. 300 bar. Directly after compression, the colour of the moist moulding is measured while still in the mould by applying the colorimeter. Measurement is made at four points on the moulding.

### Evaluation:

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The above-stated method is performed with both the pellet test piece and with the associated powder standard as reference.

The relative colour intensity of the specimens is determined to DIN 55986/A using colour intensity criterion Y.

Particle size was determined on pellets using a Malvern® Mastersizer S.

# Measurement of particle size with Malvern Mastersizer S

Two different methods, which differed with regard to the intensity of the dispersion process, were used for the dispersion of the substances under investigation. Elevated dispersion energy was achieved by using an ultrasound sonotrode with an input power of 200 W. A ®Labsonic U model ultrasound sonotrode from B. Braun Diessel Biotec was used. 500 mg of the material mixed with the stated quantity of dispersants and additives per 50 ml of distilled water were dispersed for 2 minutes using the ultrasound sonotrode. A setting of an input power of 200 W is used, with the sonotrode impulse controller being set to 0.5. Beaker-type containers having a diameter of approx. 45 mm and a height of 55 mm are used. The sonotrode is immersed approx. 2 cm into the suspension, with care being taken to ensure that the distance from the container walls is as uniform as possible. The entire suspension is introduced into the measurement chamber of the instrument and stirred and pumped at 50% of maximum power during the measurement. An alternative method of achieving low dispersion energies is to use the measuring instrument's internal ultrasound chamber. In this case, 50 mg of the substance under investigation, optionally also together with the stated additives, are placed into the instrument's ultrasound chamber, which is full of distilled water. Ultrasound power is set to 70%; the duration of dispersion is 2 minutes; the other settings remain unchanged. The

values stated are the mean of the distribution by volume (D[4.3]) and/or the 10, 50 and 90% percentile values of the distribution by volume (D[v,0.1], D[v,0.5] and D[v,0.9]).

### 5 Screen analysis using air jet screening machine

Equipment: 250 µm DIN VA screen (DIN-ISO 3310)

Air jet screening machine: Rhewum model LPS 200 MC (4 mm

nozzle, 18 rpm, 35 m<sup>3</sup>/h air, screening time 1 minute)

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Method: 20 g of the specimen to be analysed are placed on the screen and then

screened with the screening machine.

Once screening is complete, the weight of the oversize is determined

and the percentage fraction thereof relative to the initial weight

calculated.

### Draining behaviour:

Draining behaviour is determined to DIN 53 211 using a drain cup

20 (100 ml volume, 6 mm drain nozzle).

The following Examples describe the invention without limiting it.

### Test series 1

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®Flammruß 101 (lampblack, commercial product of Degussa) was vigorously mixed in a plough bar mixer (from Lödige, Paderborn) with various quantities of polyethylene glycol ®PEG 400 (commercial product of EC-Dormagen) and ammonium lignin sulfonate (commercial product of Lignotec, Düsseldorf).

These mixtures were compacted in up to 3 compacting operations or also degassed in a vacuum press and compacted in a single stage using a model CS25 unit from Bepex, Leingarten.

5 The material was coarse-ground in a model MGI 314 coarse grinder from Frewitt with a 1.5 mm mesh screen and separated from the fines fraction using a model 
®Conflux 323 μm mesh screen from Siebtechnik, Mühlheim. Table 1 below summarises the test conditions and results. Free-flowing, non-dusting, irregularly shaped pellets having an elevated relative colour intensity were obtained.

Test series 2

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Mixtures of Flammruß 101 were produced as in test series 1 with differing quantities of auxiliary substances, degassed in a vacuum press and compacted using compactor CS 25 and a precompaction screw with a strongly tapered section. Table 2 summarises the test conditions and results. Where auxiliary substances were used, stable pellets having improved relative colour intensity were obtained.

As the quantity of auxiliary substances increases, not only do the compressive forces established increase after the compression operation, but so too do bulk density, yield, stability and relative colour intensity (Table 2).

### Comparative Tests

25 At very low compressive forces or without auxiliary substances, bulk densities remain low, wherein yield and relative colour intensity are lower (Table 2).

# Table 1

	Additive	Compressive Bulk force densit	Bulk density	Relative colour intensity in	Particle size distribution	Pycnometric density/bulk	Throughput (kg/h)
		(kN/cm)	(g/cm³)	concrete**) (%)	D(4.3)	density	
Powder			0.18		3.90	10.1	
Pellet, 1st pass	2.5% PEG/2.5% ALS	5	0.25	93		7.1	43
Pellet, 2nd pass	2.5% PEG/2.5% ALS	18	0.34	117	0.78	5.4	7.5
Pellet, 3rd pass	2.5% PEG/2.5% ALS	24	0.40	126	99.0	4.6	92
Pellet, 1st pass	10% PEG/2.5% ALS	6	0.33	113	-	5.5	53
Pellet, 2nd pass	10% PEG/2.5% ALS	25	0.46	125	0.73	4.0	132
Pellet*	10% PEG/2.5% ALS	23	0.49	123	92.0	3.7	68
Pellet*	10% PEG/5% ALS	23	0.46	123	0.73	4.0	82

\* previously degassed with ®Vacupress

PEG = polyethylene glycol

ALS = ammonium lignin sulfonate

\*\* measured against powder mixture



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# CONTROL CONTROL

Table 2

	Additive	(%)	Compressive force (1)	Compressive Throughput force (1) CS 25	Bulk density	Pycnometric Over density/bulk size	Over- size	Drain time (s)	Pycnometric Over- Drain time Relative colour Stability (2) Particle size density/bulk size (s) intensity in distribution	Stability (2)	Particle size distribution
			(kN/cm)	(kg/h)	(g/cm³)	density	(%)		concrete (%)		D(4.3) µm
Powder		L			0.18				100		3.90
Pellet 1	10% PEG/	15	20	143	0.43	4.2	50.2	33	137	er.	0.82
Dallot 7	576 ALS 6 79, DEG/	2	14	133	0.38	8 4	45.3	34	143	3	0.77
7 121	3.3% ALS	2		}	2	}					
Pellet 3	3.4% PEG/	'n	13	84	0.33	5.5	42.6	33	129	2	0.74
	1.6% ALS										
Pellet 4	2% PEG/	~	6	70	0.29	6.2	47.0	33	116	_	86'0
	1% ALS										
mparative	Comparative no additives	0	3	approx. 10**	0.25	7.3	35.6	35.6 does not flow	103	0	3.67
test											

<sup>\*\*</sup> Product flowed very irregularly.

(1) = max. compressive force (after Vacupress) in 1 pass.

(2) = sensory test, values from 0-5 (0 = soft, 5 = hard).



### Patent Claims

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- Process for the production of carbon black pellets, in which carbon blacks are compacted in the presence of auxiliary substances and comminuted to yield pellets, wherein the pellets have an average particle size of 0.3 to 2 mm, preferably of 0.5 to 1.0 mm, characterised in that the quotient of density and bulk density is between 3.0 and 10, preferably between 3.5 and 8.
- Process according to claim 1, characterised in that compaction is performed
   twice or more.
  - Process according to claims 1 and 2, characterised in that one or more binders and/or dispersants are used as auxiliary substances in a total quantity of 0.1 to 25 wt.%, preferably of 1 to 20 wt.%, in particular of 3 to 15 wt.%.

 Process according to claims 1 to 3, characterised in that a liquid binder is used from the group comprising polyols, polyethers, polyesters, oils, water and aqueous solutions of polymeric salts or molasses.

- Process according to claims 1 to 3, characterised in that a dispersant is used from the group comprising lignin sulfonates and naphthalene/formaldehyde condensation products.
- Process according to claims 1 to 5, characterised in that compressive forces
   of between 1 and 100 kN/cm, preferably of between 5 and 50 kN/cm, in particular of between 10 and 30 kN/cm are used.
  - Process according to claims 1 to 6, characterised in that compaction units are used in the form of screws, rollers, die presses or extruders.

- Process according to claims 1 to 7, characterised in that compaction is performed in two or more compaction stages at differing compressive forces.
- Process according to claim 8, characterised in that there are two compaction
   stages.
  - Process according to claims 1 to 9, characterised in that screening is performed as a subsequent stage.
- 10 11. Process according to claims 1 to 10, characterised in that the separated screen fractions are pelletised.
- Process according to claims 1 to 11, characterised in that the resultant pellets are coated with a thin layer of a material from the group comprising waxes, polyethers, polyolefins and polyvinyl alcohols.
  - Process according to claims 1 to 12, characterised in that preservatives and/or fragrances are additionally added.
- 20 14. Compacted carbon black pellets having a relative colour intensity, relative to the powder on which they are based, of greater than 100%.
  - 15. Carbon black pellets according to claim 14, characterised in that that they have an average particle size of 0.3 to 2 mm, preferably of 0.5 to 1.0 mm, and that the quotient of density and bulk density is between 3.0 and 10, preferably between 3.5 and 8.
  - Carbon black pellets according to claims 14 and 15, characterised in that they
    are obtained by compaction performed twice or more.

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- 17. Carbon black pellets according to claims 14 to 16, characterised in that they contain one or more binders and/or dispersants as auxiliary substances in a total quantity of 0.1 to 25 wt.%, preferably of 1 to 20 wt.%, in particular of 3 to 15 wt.%.
- 18. Carbon black pellets according to claim 17, characterised in that the binder(s) comprise(s) a liquid substance from the group comprising polyols, polyethers, polyesters, oils, water and aqueous solutions of polymeric salts or molasses.
- 19. Carbon black pellets according to claim 17, characterised in that the dispersant comprises a substance from the group comprising lignin . sulfonates and naphthalene/formaldehyde condensation products.

### Carbon black pellets

### Abstract

This invention relates to carbon black pellets obtained by compaction of carbon black powder in the presence of auxiliary substances, which pellets, despite the compaction, surprisingly exhibit greater colour intensity than the powders on which they are based.

### SOOT GRANULES

### ABSTRACT OF THE DISCLOSURE

This invention relates to carbon black pellets obtained by compaction of carbon black powder in the presence of auxiliary substances, which pellets, despite the compaction, surprisingly exhibit greater color intensity than the powders on which they are based.



# COMBINED DECLARATION AND POWER OF ATTORNEY ATTORNEY DOCKET NO

As a below named inventor, I hereby declare that:

My residence, post office address and citizenship are as stated below next to my name. I believe I am the original, first and sole inventor (if only one name is listed below) or an original, first and joint inventor (if plural names are listed below) of the subject matter which is claimed and for which a patent is sought

on the invention entitled

### "SOOT GRANULES"

the specification of which is attached hereto,

or was filed on March 10, 1999

as a PCT Application Serial No. PCT/EP99/01537

I hereby state that I have reviewed and understand the contents of the above-identified specification, including the claims.

I acknowledge the duty to disclose information which is material to the patentability of this application in accordance with Title 37, Code of Federal Regulations, \$1.56.

I hereby claim foreign priority benefits under Title 35, United States Code, \$119 of any foreign application(s) for patent or inventor's certificate listed below and have also identified below any foreign application for patent or inventor's certificate having a filing date before that of the application on which priority is claimed:

Prior Foreign Application(s), the priority(ies) of which is/are to be claimed:

198 11 553.9 (Number) Germany (Country)

March 18, 1998 (Month/Day/Year Filed)

I hereby claim the benefit under Title 35, United States Code, \$120 of any United States application(s) listed below and, insofar as the subject matter of each of the claims of this application is not disclosed in the prior United States application in the manner provided by the first paragraph of Title 35, United States Code, \$112, I acknowledge the duty to disclose the material information as defined in Title 37, Code of Federal Regulations, \$1.56 which occured between the filling date of the prior application and the national or PCT international filling date of this application:

(Application Serial No.)	(Filing Date)	(Status) (patented, pending, abandoned)
(Application Serial No.)	(Filing Date)	(Status)
		(patented, pending, abandoned)

I hereby declare that all statements made herein of my own knowledge are true and that all statements made on information and belief are believed to be true; and further that these statements were made with the knowledge that willful false statements and the like so made are punishable by fine or imprisonment, or both, under Section 1001 of Title 18 of the United States Code and that such willful false statements may jeopardize the validity of the application or any patent issued thereon.

POWER OF ATTORNEY: As a named inventor, I hereby appoint the following attorney(s) and/or agent(s) to prosecute this application and to transact all business in the Patent and Trademark Office connected therewith:

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